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# The Chemist Analyst

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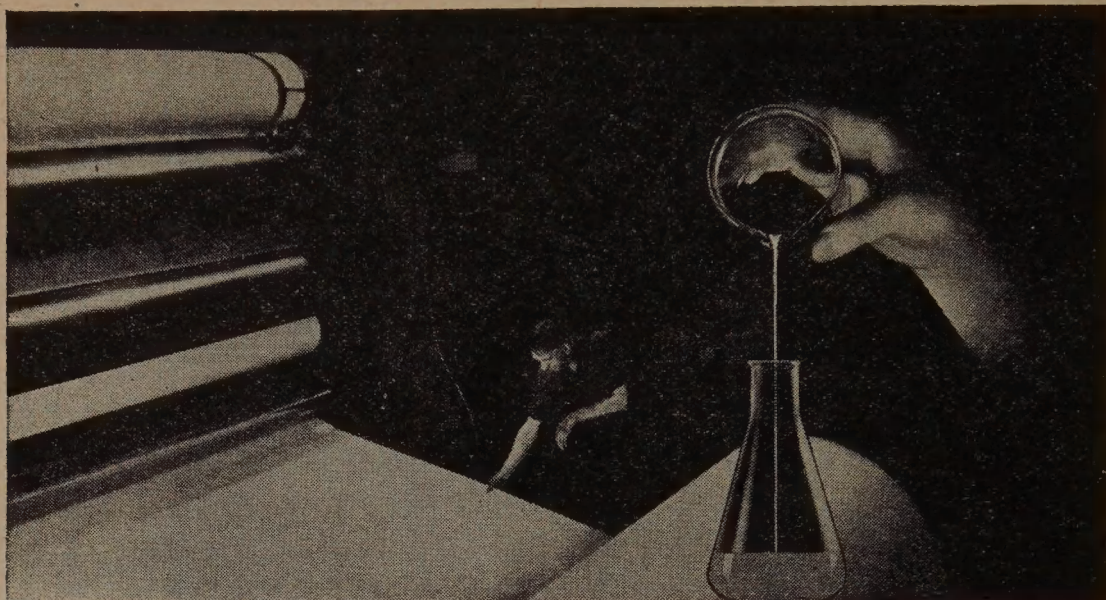
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S. B. WILDRICK, *Editor*

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## D.D.T.

D.D.T.'s dramatic wartime debut, its spectacular results in the realm of the military and its intriguing promise for civilian application are no longer headline news. Yet rarely in the memory of the American chemical industry has a chemical product so captivated the public imagination and aroused the serious interest of the scientist.

As a chemical it was synthesized in 1874 by Othmar Zeidler, a student at Strasbourg. It reposed in the scientific archives, as just another chemical of no demonstrated economic value, until 1939. By some fortuitous circumstance it was then investigated as a possible means of combating a disastrous invasion of the potato beetle in Switzerland. Alert to its wartime potentialities, the United States Department of Agriculture conducted intensive investigations. In 1942 D.D.T.'s lousicidal properties were revealed.

Here was the weapon to deal with the body louse, the carrier of the dreaded typhus! The disease had already appeared in some areas of the European theatre of war. The American chemical industry rallied to the call for D.D.T. sounded by the Surgeon General's Office. Several chemical manufacturers, under license from J. R. Geigy A.-G., patriotically responded with large scale production. Within the space of a few months' time Allied soldiers and liberated peoples were enjoying the beneficent protection of this remarkable insecticide. Countless stories of D.D.T.'s vital role in warfare, related on the air and in print, have vividly impressed the public. So much so that today D.D.T. is now a household word, although few civilians have ever seen it.

Chemically D.D.T. is known as 2,2-bis(*p*-chlorophenyl)-1,1,1-trichloroethane. It is produced by the reaction of chloral and chlorobenzene with oleum to form dichloro-diphenyl-trichloroethane, its isomers and polymers. It is a fine powder having a faint floral odor. It is white to cream in color. It is insoluble in water. It is soluble in varying degrees in such solvents as cyclohexanone, benzene, toluene, xylene and numerous other organic solvents, including petroleum oils and vegetable oils. Its inherent toxicity to humans and warm-blooded animals is dependent upon many factors, such as physical state (solution, emulsion or powder) and the relative concentration in each instance (2). Similarly the mode of entrance into the body, whether by oral admission, inhalation or skin absorption, is of toxicological concern. Much more work remains to be done in evaluating this product's toxic properties.

D.D.T. is now being prepared in two grades: the technical powder and the aerosol grade. The bulk of present production is devoted to the technical

(continued on page 71)



## A Ferrocyanide-Cerimetric Method for the Determination of Zinc in Ores

J. P. MEHLIG and J. K. CLAUSS \*

Oregon State College, Corvallis, Ore.

There has long been a real need for a rapid, reliable, titrimetric method for the determination of zinc. While the Waring method (9) involving a titration with potassium ferrocyanide solution has been widely used, it has been the subject of much controversy in regard to the experimental conditions and the accuracy of the results.

The purpose of the work described in this paper was to develop a method for zinc in ores, based upon the precipitation of potassium zinc ferrocyanide by an excess of standard potassium ferrocyanide solution and titration of the excess with standard ceric sulfate solution. This work was the basis of a thesis submitted in May 1943 by the junior writer (1) to the Graduate School of Oregon State College for the degree of Master of Science. Recently a similar method has been reported by Miller, Boyle and Neill (6) for zinc in magnesium alloys. They worked with much smaller amounts of zinc, used a different indicator, and instead of removing iron tied it up as ferric ferrocyanide. This procedure for iron causes a slight negative error for zinc and would not be feasible for ores in which the percentage of iron is normally much higher than in magnesium alloys.

### Preparation of Solutions

*Potassium Ferrocyanide.* An approximately 0.1 *M* solution was made by dissolving the trihydrate in water. This solution was standardized by the ferrocyanide-cerimetric method described below against a sphalerite ore, the zinc content of which was determined gravimetrically by weighing zinc ammonium phosphate (5). One milliliter of this solution was found to be equivalent to 0.009961 gram of zinc.

*Ceric Sulfate.* Eighty grams of ceric ammonium sulfate dihydrate were dissolved in 500 ml. of 1 *M* sulfuric acid and the solution was diluted to 1000 ml. and standardized against the standard potassium ferrocyanide solution with sodium diphenylamine sulfonate as indicator. Each milliliter was equivalent to 0.9108 ml. of the ferrocyanide solution.

*Sodium Diphenylamine Sulfonate.* A 0.01 *M* solution was made by dissolving 0.32 gram of the barium salt in 100 ml. of water, adding 0.5 gram of sodium sulfate and filtering off the barium sulfate.

### Determination of Zinc

A 1-gram sample of the ore was warmed in a covered casserole with a mixture of 15 ml. each of concentrated hydrochloric and nitric acids. When action had ceased the mixture was evaporated to a syrupy consistency, transferred to a 250-ml. beaker, diluted to 100 ml., and heated nearly to boiling. Ammonium hydroxide was added in excess of that required for complete precipitation of ferric hydroxide. After the precipitate had settled, the supernatant liquid was filtered and the residue was washed by decantation with

\* Present address: General Chemical Co., Edgewater, N. J.



five 15-ml. portions of hot water, transferred to the filter and thoroughly washed with hot water. The filtrate was neutralized with 6 *M* hydrochloric acid until the precipitate of zinc hydroxide which formed had redissolved. Ten milliliters more of the acid were added and the volume of the solution was adjusted to about 150 ml. To the cold solution 30 ml. of standard potassium ferrocyanide solution were added dropwise from a burette with stirring. After the precipitate of potassium zinc ferrocyanide had settled for 15 minutes, it was filtered on a Gooch crucible provided with an asbestos mat of 4 to 6 mm. thickness, using 0.3 *M* hydrochloric acid for transferring. The residue was finally washed with four 15-ml. portions of this dilute acid. The excess potassium ferrocyanide in the filtrate was titrated in the suction flask with standard ceric sulfate solution with 8 drops of sodium diphenylamine sulfonate solution as indicator.

### Results

The results given by four sphalerite ores are shown in Table I, which also includes for comparison the values obtained by the gravimetric method (5) in which zinc ammonium phosphate was weighed. The former do not vary from the latter by more than 0.03 per cent. The percentage error ranges from 0.0 to 0.2 per cent compared to a range of 1 to 6 per cent for the Miller, Boyle and Neill method (6). Results were duplicated with a precision of less than 0.10 per cent and an average deviation of 1.7 parts per thousand.

### Discussion

Since the relation between potassium ferrocyanide and zinc is an empirical one, the ferrocyanide solution must be standardized against zinc following the same procedure as for the determination. That the composition of the zinc precipitate is constant under the conditions is shown by the low average deviation of the results of the ferrocyanide-cerimetric determinations and by the very close agreement of these results with those of the gravimetric determinations.

TABLE I. RESULTS OBTAINED BY THE FERROCYANIDE-CERIOMETRIC METHOD

| Sample No. | Zinc Found by Ferrocyanide-Cerimetric Method | Zinc Found by Gravimetric Method | Deviation |
|------------|----------------------------------------------|----------------------------------|-----------|
| 1          | 14.34 %                                      | 14.34 %                          | 0.00 %    |
| 2          | 16.57                                        | 16.60                            | - 0.03    |
| 3          | 18.28                                        | 18.29                            | - 0.01    |
| 4          | 19.81                                        | 19.81                            | 0.00      |

It is necessary to remove the zinc precipitate by filtration before the titration, since it is slowly oxidized by the ceric sulfate. The formation of the precipitate is facilitated by the presence of ammonium salts, of acid and of excess potassium ferrocyanide (3, 4, 8).

If the sample contains much silica it is advisable to remove it by filtration before precipitation of ferric hydroxide, and, if the latter precipitate is heavy, it should be separated by double precipitation to prevent occlusion of zinc (9).

Iron, copper, antimony, manganese and aluminum interfere just as in the Waring method (9). Aluminum is removed as hydroxide along with iron.



Miller, Boyle and Neill (6) recommend the removal of copper with test lead. Cadmium, which so often occurs in zinc ores, does not interfere since its ferrocyanide is soluble in hydrochloric acid.

A complete determination can be made in less than an hour after solution of the sample. The results compare very favorably with those obtained gravimetrically and have greater precision than those given by the Waring titrimetric method (2, 7). The end point color change is also much sharper and more easily detected. The disadvantages are that two standard solutions are required and that the zinc precipitate must be filtered.

### Summary

A titrimetric method has been developed for the determination of zinc in ores which consists in precipitating potassium zinc ferrocyanide with excess standard potassium ferrocyanide solution and titrating the excess with standard ceric sulfate solution. The method gives easily reproducible results which check very closely with those given by the zinc ammonium phosphate gravimetric method. The precision is much superior to that of the Waring ferrocyanide titrimetric method.

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## A Simple Method for the Conversion of Fractional pH Values

D. M. BATSON and FRANCES O. BATSON  
New Orleans, Louisiana

In chemical work it is frequently necessary to convert observed pH readings to terms of hydrogen-ion concentration. For cases in which pH is determined or recorded only to the nearest tenth of a pH unit, the use of the principles herein discussed should prove valuable in quickly effecting the desired conversion. Also, these principles may perhaps be utilized advantageously in assisting laboratory workers or students to better understand the relationship between fractional pH values and equivalent hydrogen-ion concentrations.



According to definition, pH values indicate degree of acidity or alkalinity in terms of the logarithm of the reciprocal of the hydrogen-ion concentration in gram-equivalents of ionic hydrogen per liter of solution. The highest degree of acidity commonly represented on the pH scale is pH 0.00 and the lowest degree of acidity (or highest degree of alkalinity) is pH 14.00, corresponding to hydrogen-ion concentrations of  $1.0080 \times 10^0$  grams per liter and  $1.0080 \times 10^{-14}$  grams per liter respectively. (In practical usage the significant figures to the right of the zeros in the decimal fraction are dropped.) Thus, it is well known that the hydrogen-ion concentration is  $1 \times 10^0$  grams per liter at pH 0,  $1 \times 10^{-1}$  at pH 1,  $1 \times 10^{-2}$  at pH 2, and so on; or in other words, that a decrease in pH of 1.0 unit means a ten-fold increase in hydrogen-ion concentration.

A fact which is less commonly realized is that a decrease in pH (increase in acidity) of 0.30 unit corresponds almost exactly to a doubling of the hydrogen-ion concentration and, conversely, an increase in pH of 0.30 unit corresponds to a halving of the hydrogen-ion concentration. The validity and usefulness of this relationship will become apparent after a few calculations, as follows: Using the conventional symbol  $[H^+]$  to represent hydrogen-ion concentration in gram-equivalents per liter of solution, then

$$\text{since by definition:} \quad \log \frac{1}{[H^+]} = \text{pH} \quad (1)$$

$$\text{it follows that:} \quad \frac{1}{[H^+]} = \text{antilog pH} \quad (2)$$

$$\text{And:} \quad [H^+] = \frac{1}{\text{antilog pH}} \quad (3)$$

By following equation (3) and using a table of antilogarithms, the  $[H^+]$  equivalents of various pH values at one-tenth unit intervals between pH 0.0 and pH 1.0 were obtained, as recorded in Table I.

TABLE I

| pH  | $[H^+]$                |
|-----|------------------------|
| 1.0 | $1.00 \times 10^{-1}$  |
| 0.9 | $1.26 \times 10^{-1}$  |
| 0.8 | $1.58 \times 10^{-1}$  |
| 0.7 | $2.00 \times 10^{-1}$  |
| 0.6 | $2.51 \times 10^{-1}$  |
| 0.5 | $3.16 \times 10^{-1}$  |
| 0.4 | $3.98 \times 10^{-1}$  |
| 0.3 | $5.01 \times 10^{-1}$  |
| 0.2 | $6.31 \times 10^{-1}$  |
| 0.1 | $7.94 \times 10^{-1}$  |
| 0.0 | $10.00 \times 10^{-1}$ |



The foregoing data show that, with a decrease in pH (increase in acidity) of 0.3 pH unit,  $[H^+]$  approximately doubles; thus, in going from pH 1.0 to pH 0.7 or from pH 0.7 to pH 0.4, a doubling of  $[H^+]$  occurs. Proceeding on this basis it may be shown that, if  $[H^+]$  corresponding to each of any three successive pH values in Table I is committed to memory,  $[H^+]$  for the remaining pH values can be easily derived therefrom without further recourse to a table of antilogarithms. For example, assume that  $[H^+]$  is known only for pH 1.0, pH 0.9 and pH 0.8. Then at pH 0.7, which is 0.3 pH below (more acid than) pH 1.0,  $[H^+]$  should be just double that at pH 1.0, or  $2.00 \times 10^{-1}$ . Continuing this procedure,  $[H^+]$  at pH 0.6 should be twice that at pH 0.9, or  $2.52 \times 10^{-1}$ . It will be seen that these derived values for  $[H^+]$  are in close or perfect agreement with the  $[H^+]$  values listed in Table I, and that  $[H^+]$  values corresponding to the successively lower pH values listed in the table may be similarly obtained.

In order to further extend the usage of this method so as to encompass the entire pH scale at intervals of 0.1 pH unit, it is only necessary to properly change the numerical value of the negative exponent in the  $[H^+]$  values recorded in Table I. Since a decrease in pH of 1.0 is equivalent to a ten-fold increase in  $[H^+]$ , each increase in pH of 1.0 means that  $[H^+]$  is decreased to one-tenth of its original magnitude. That each such ten-fold decrease in  $[H^+]$  may be represented by an increase of *one* in the negative exponent of the  $[H^+]$  expression is illustrated by a statement made earlier, namely, that  $[H^+]$  is  $1.00 \times 10^{-1}$  at pH 1.0,  $1.00 \times 10^{-2}$  at pH 2.0,  $1.00 \times 10^{-3}$  at pH 3.0, etc. The same holds true for fractional pH values; thus, remembering that  $[H^+]$  is  $1.58 \times 10^{-1}$  at pH 0.8, it is obvious that  $[H^+]$  will be  $1.58 \times 10^{-2}$  at pH 1.8,  $1.58 \times 10^{-3}$  at pH 2.8, and so on. For further convenience it is worthwhile to note that the negative exponent in any  $[H^+]$  expression is always numerically equal (except for sign) to one plus the whole number of the corresponding pH value; *e. g.*, in the case of pH 2.8 above, at which  $[H^+]$  is  $1.58 \times 10^{-3}$ , the whole pH number is 2, and 1 plus 2 is the numerical value of the exponent. (This "rule of thumb" holds valid except when the whole number of the pH value is followed by a zero, such as pH 1.0, 2.0, etc., in which case the numerical value of the negative exponent is the same as the whole number of the pH value.)

To summarize the preceding paragraphs, it has been shown that if  $[H^+]$  values corresponding to pH 1.0, pH 0.9 and pH 0.8 are committed to memory, the  $[H^+]$  equivalent of the other pH values (at intervals of 0.1 pH unit) between pH 1.0 and pH 0.0 can be quickly computed therefrom without reference to logarithm tables. It was further shown that from these  $[H^+]$  values the  $[H^+]$  at any other higher pH can be easily found by changing only the negative exponent of the appropriate  $[H^+]$  expression.

As a final example of the application of these principles, suppose that we wish to obtain the  $[H^+]$  equivalent of pH 7.5. First, find  $[H^+]$  at pH 0.5 by noting that pH 0.5 is 0.30 unit more acid than pH 0.8 and recalling that since  $[H^+]$  is  $1.58 \times 10^{-1}$  at pH 0.8 then  $[H^+]$  at pH 0.5 must be twice as great, or  $3.16 \times 10^{-1}$ . Now, changing only the exponent of the latter expression as in foregoing examples, we see that at pH 7.5  $[H^+]$  will be  $3.16 \times 10^{-8}$ .



## Determination of Iron in High Silicon-Aluminum-Base Alloys Copper Powder Reduction Method

LOUIS SILVERMAN, ANITA YUNKER and ANNE GEARING  
Pittsburgh, Penna.

The procedure most used for the determination of iron in aluminum alloys is that in which the aluminum is dissolved in sodium hydroxide and the iron is left in the residue with many other elements. If the alloy contains a high percentage of silicon, a large quantity of silica will remain with the iron. The iron may now be dissolved in acid, or the silica may be volatilized with hydrofluoric acid and then the resulting solution prepared for the iron titration. This procedure is somewhat long and the precision of results is not satisfactory, except in experienced hands.

It is observed that a silicon (50 per cent) aluminum alloy easily dissolves in sodium hydroxide solution and that subsequent acidification and oxidation give a clear solution. At this point the iron may be reduced by copper powder (1) and the iron determined by permanganate titration.

### Proposed Procedure

Weigh 2 grams of aluminum base alloy and transfer to a tall form 300 ml. beaker. Add 25 ml. of 15 per cent sodium hydroxide solution. (Add water at once if the reaction is too rapid.) Allow the beaker to cool. Dilute with water to 75 ml. Drop in a small piece of Congo Red paper and add sulfuric acid (1:1) until the solution is acid (usually 20 to 25 ml.). From a graduate add an additional 20 ml. of sulfuric acid (1:1).

Add slowly and with stirring a solution of potassium permanganate (about 0.1 normal) until the dark solution clears and the permanganate color will not fade for at least ten seconds (5 to 25 ml.).

Add about 0.4 gram of copper powder and stir for several minutes. Add a small portion of paper pulp and filter through an 11 cm. No. 41 Whatman paper. Wash four times with water. Add 5 ml. of sirupy phosphoric acid. Add a piece of ice. Titrate slowly with standard 0.1 N permanganate. The end-point is sharp and should not fade within sixty seconds.

### Results

In Table I are listed the percentages of iron found in various types of aluminum base alloys, including many that are low in silicon. Semicolons separate the results of the individual authors. The values enclosed in parentheses are those given by the manufacturers.



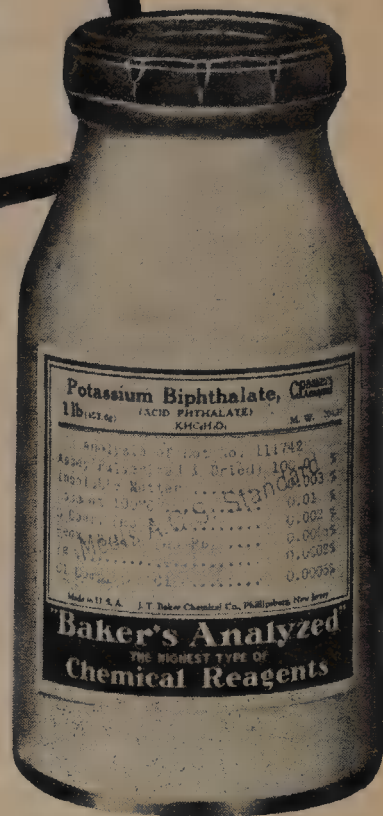
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TABLE I

|    | Percentage of Fe |           | Si   | Cu    | Ti   | Mn   | Zn   |
|----|------------------|-----------|------|-------|------|------|------|
|    | Authors          | Others    |      |       |      |      |      |
| 1  | 1.49, 1.50; 1.53 | (1.53)*   | —    | —     | —    | —    | —    |
| 2  | 0.40; 0.44       | (0.395)** | —    | —     | —    | —    | —    |
| 3  | 0.42; 0.42       | (0.46)    | 50   | 0.05  | 0.04 | —    | —    |
| 4  | 0.35             | (0.46)    | 50   | 0.01  | 0.04 | —    | —    |
| 5  | 0.27             | (0.31)    | 50   | 0.02  | 0.05 | —    | —    |
| 6  | 0.32             | (0.37)    | 48   | 0.02  | 0.03 | —    | —    |
| 7  | 0.34, 0.32       | 0.32†     | 50   | —     | —    | —    | —    |
| 8  | 0.28; 0.29       | (0.56)    | 49   | 0.01  | 0.03 | —    | —    |
| 9  | 0.35             | (0.38)    | 47   | 0.01  | 0.03 | —    | —    |
| 10 | 0.21; 0.25       | (0.39)    | 48   | 0.01  | 0.03 | —    | —    |
| 11 | 0.28; 0.28       | (0.76)    | 45   | 0.02  | 0.03 | —    | —    |
| 12 | 0.19; 0.20       | (0.43)    | 49   | 0.02  | 0.04 | —    | —    |
| 13 | 1.56             | (1.54)‡   | 5.0  | —     | —    | —    | —    |
| 14 | 1.54, 1.54       | —         | —    | 6.72  | —    | —    | —    |
| 15 | 1.54, 1.54       | —         | —    | 6.85  | —    | —    | —    |
| 16 | 1.54, 1.54       | —         | —    | 6.94  | —    | —    | —    |
| 17 | 1.49; 1.46       | —         | —    | 6.86  | —    | —    | —    |
| 18 | 1.34; 1.30       | (1.34)‡   | 5.0  | —     | —    | —    | —    |
| 19 | 1.32; 1.32       | —         | low  | 4.04  | —    | —    | —    |
| 20 | 1.26, 1.26       | —         | low  | 4.42  | —    | —    | —    |
| 21 | 1.23, 1.26       | —         | 2.12 | 7.29  | —    | 0.11 | —    |
| 22 | 1.23, 1.23       | (1.14)‡   | 5.0  | —     | —    | —    | —    |
| 23 | 1.14             | 1.06†     | 5.9  | 0.11  | 0.13 | —    | —    |
| 24 | 1.15             | 1.20†     | 2.56 | 4.47  | 0.05 | —    | —    |
| 25 | 1.13             | 1.23†     | 2.60 | 4.4   | 0.05 | —    | —    |
| 26 | 1.06; 1.02       | (0.95)‡   | —    | —     | —    | —    | —    |
| 27 | 1.06; 1.06       | (1.02)‡   | 2.8  | 16.02 | —    | 0.10 | 1.45 |
| 28 | 1.06; 1.02       | (1.02)‡   | 2.8  | 16.02 | —    | 0.10 | 1.45 |
| 29 | 1.02             | 1.11†     | 2.5  | —     | 0.05 | —    | —    |
| 30 | 0.97             | 0.95†     | 5.5  | 0.08  | 0.14 | —    | —    |
| 31 | 0.87, 0.90       | —         | low  | 1.34  | —    | —    | —    |
| 32 | 0.89             | 0.88†     | 5.6  | 0.11  | 0.13 | —    | —    |
| 33 | 0.83             | (0.83)    | 5.4  | 0.09  | 0.13 | —    | —    |
| 34 | 0.73, 0.75       | —         | 5.4  | 0.18  | —    | 0.16 | —    |

\* Bureau of Standards Sample No. 86b.

\*\* Bureau of Standards Sample No. 85.

‡ Zimmermann-Reinhardt method.

† Spectrographic method.



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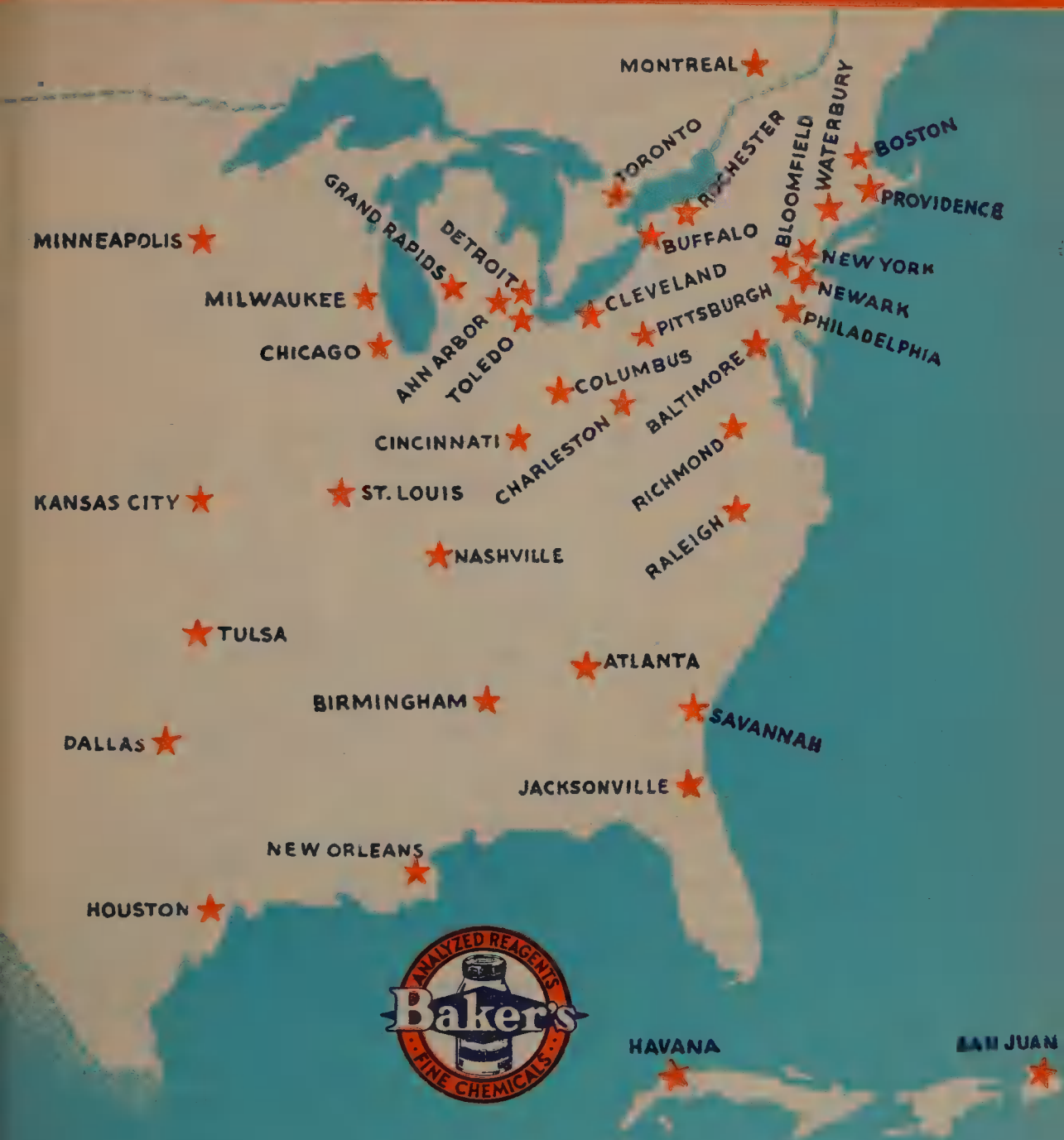
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|    | Percentage of Fe |        | Si   | Cu   | Ti | Mn   | Zn |
|----|------------------|--------|------|------|----|------|----|
|    | Authors          | Others |      |      |    |      |    |
| 35 | 0.70, 0.73       | —      | —    | —    | —  | —    | —  |
| 36 | 0.68, 0.68       | —      | —    | —    | —  | —    | —  |
| 37 | 0.64, 0.67       | —      | 11.3 | 0.24 | —  | —    | —  |
| 38 | 0.64, 0.67       | —      | 1.34 | 4.16 | —  | 0.13 | —  |
| 39 | 0.64, 0.64       | —      | 8.94 | 3.14 | —  | —    | —  |
| 40 | 0.61, 0.68       | —      | —    | —    | —  | —    | —  |
| 41 | 0.59, 0.56; 0.58 | —      | —    | —    | —  | —    | —  |
| 42 | 0.56, 0.55, 0.56 | —      | —    | —    | —  | —    | —  |
| 43 | 0.53, 0.53       | —      | —    | —    | —  | —    | —  |
| 44 | 0.50, 0.53       | —      | —    | —    | —  | —    | —  |
| 45 | 0.45, 0.50       | —      | 1.69 | —    | —  | 0.26 | —  |
| 46 | 0.38, 0.40       | —      | 4.65 | 1.20 | —  | —    | —  |
| 47 | 0.37, 0.41       | —      | —    | 4.62 | —  | 0.82 | —  |
| 48 | 0.36, 0.36       | —      | 2.07 | 3.28 | —  | —    | —  |
| 49 | 0.33, 0.33       | —      | —    | 1.12 | —  | —    | —  |
| 50 | 0.32, 0.32       | —      | 0.33 | 3.28 | —  | —    | —  |
| 51 | 0.29, 0.30       | —      | 0.08 | —    | —  | —    | —  |

### Discussion

The procedure outlined is quite simple. Only one filtration is required namely that of the excess copper. As to time, a set of ten to twelve irons can be run within two to three hours; in fact, the time elapsed between acidification of the caustic solution and the filtration must not be over one hour, else silicic acids will clog the filter papers and make filtration slow or impossible.

In tracing the chemical changes of the elements present, aluminum is converted to sodium aluminate then to aluminum sulfate; silicon is dissolved as silicate then acidified to the acid; some copper is converted to sulfate during the preliminary oxidation; iron is dissolved by the sulfuric acid, oxidized by permanganate, reduced by copper powder and quantitatively determined by standard permanganate; some manganic oxides are formed during the caustic reaction but all are reduced subsequently in the presence of acid and ferrous iron; tin is oxidized during the preliminary addition of permanganate and not reduced by copper; magnesium and zinc do not interfere; and in another paper (2) it was shown that titanium is without effect.

The function of the preliminary oxidation is the oxidation of tin, titanium and any silico-organic compounds which might reduce permanganate. The dark colored solution formed after acidification by sulfuric acid is clarified by the permanganate, although some of the copper chips) high copper alloys) may remain and be filtered off with the copper powder.

### Acknowledgements

The authors wish to thank Richard Scheideman and Robert Eberly for contributing spectrographic values, also James Reamer for check analyses on many samples.

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## Evaluation of Liquid Soaps

D. H. MATHESON

Hamilton, Ontario, Canada

The determination of the water softening power of liquid soaps provides an easy and reliable method of estimating the relative value of the products of different manufacturers. The water softening power may be defined as the volume of water of specified hardness which is completely softened by a unit volume of the soap. This is a significant property of liquid soaps, as water softening power is actually what is being purchased.

Since the hardness of the local water may vary from time to time, and also since some difficulty may be experienced in determining the endpoint when both calcium and magnesium are present, the use of a synthetic hard water of approximately the same hardness as the local water is advantageous.

**Preparation of Standard Hard Water.** Weigh exactly 1.000 g. of pure dry calcium carbonate and transfer it to a 250 ml. beaker containing 50 ml. of distilled water. Add 10 ml. of HCl (1:1) and evaporate to dryness to remove excess acid. Dissolve the residue in distilled water and dilute to 1000 ml. This water has a hardness of 1000 p.p.m. of  $\text{CaCO}_3$ . Dilute as required to approximate the hardness of the local water. (For example, dilution to 250 p.p.m. would be satisfactory if the tap water hardness were 225 to 275 p.p.m.)

**Titration.** Pipette 10 ml. of the well mixed soap sample to a volumetric flask and dilute with distilled water to 100 ml. Fill the burette with this solution.

Measure 100 ml. of the standard hard water into a 250 ml. glass stoppered bottle. Titrate with the diluted soap solution, adding it at first in 0.5 ml. portions and finally in 0.1 ml. portions, shaking vigorously between additions until a foam is obtained that is stable for one minute. The number of milliliters of the soap solution required, divided by 10, is the volume of the original sample required to soften 100 ml. of the standard hard water, from which the volume of water softened by 1 ml. of the soap sample is calculated.

Table I shows the results obtained with three representative soap samples, using water of 100 p.p.m. hardness as the standard. The percentage of anhydrous soap was determined from the volume of fatty acids by the method of Matthews (1) and shows good correlation with the softening power.

TABLE I

| Soap Sample  | Water Softening Power | Anhydrous Soap |
|--------------|-----------------------|----------------|
| 1 (pre-war)  | 250                   | 34.5 %         |
| 2 (war-time) | 120                   | 17.0           |
| 3 (war-time) | 101                   | 14.5           |

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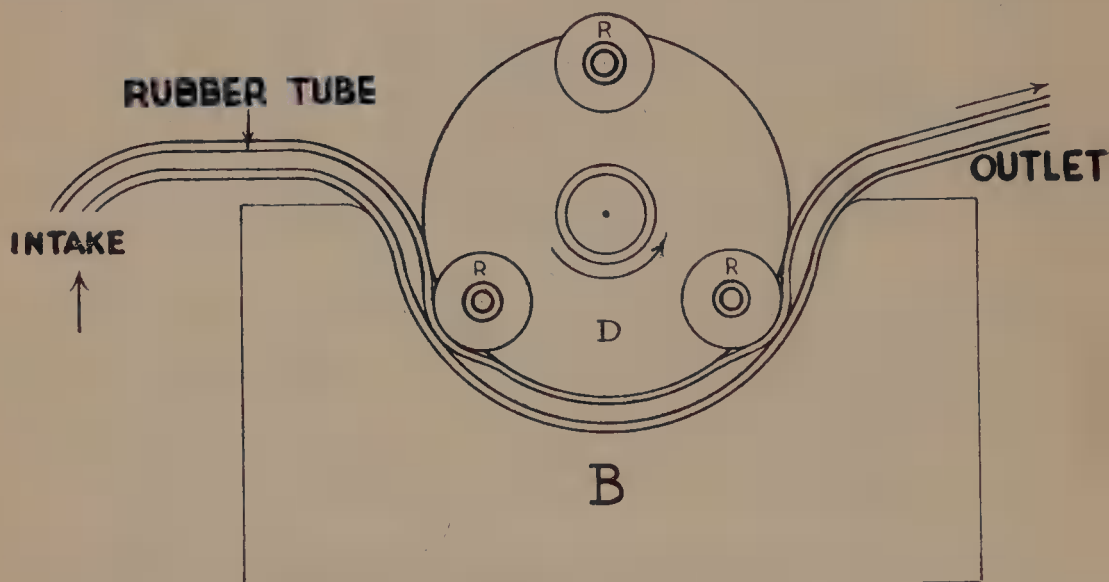


# LABORATORY SUGGESTIONS

## A Useful Pump

GEORGE W. PAWEL  
Norris, Tenn.

The accompanying sketch is an illustration of an easily made and effective pump, especially suitable for small applications or those in which the transfer of corrosive liquids is involved. No claim for originality of the idea is made.



The drawing requires little explanation. The steel disc, *D*, mounts three small rollers, *R*, firmly attached at equidistant points on its periphery. The disc revolves in the semicircular groove or well in the wooden block, *B*. The radius of the well is slightly greater than that of the disc in order just to accommodate the rubber tube. One end of the tube is sunk in the supply reservoir and the flexing of the tube under the rollers ejects the liquid from the discharge end, creating at the same time the vacuum which draws in an equal amount from the source of supply. The delivery of liquid is, of course, proportional to the speed of rotation of the disc, so that a variable speed motor for driving it would be desirable. The applications for such a cheap, simple and useful gadget in shop or laboratory are manifest.

## An Improvised Pycnometer

GEORGE VANDER VEEN

Bound Brook, New Jersey

A simple inexpensive Babcock milk test bottle may be used to replace more expensive types of pycnometers in the determination of the specific gravity of certain liquids without appreciable sacrifice of accuracy.

Requirements are: (a) that the liquid sample be sufficiently fluid to pass through the bottle neck with ease; (b) that at least 50 ml. of sample are available; (c) that a constant temperature bath with basket or tray of suitable depth is available.

The standard Babcock milk test bottle ( $8\frac{1}{2}$  inches high, 18 gram sample capacity) has a bulb capacity of about 45 ml. The neck is graduated into 80 minor divisions of 0.02 ml. each. The empty bottle weighs about 30 grams and may be weighed on the pans of most analytical balances without interference of bows, etc.

For specific gravity determinations, first determine the capacity of the Babcock bottle. Fill the previously weighed empty bottle with freshly boiled and cooled distilled water to any point along the graduated neck. Support the bottle in the constant temperature bath, maintained at the desired temperature (say 20° C.), for 30 minutes with the bath level well up along the neck. Then note the water level in the bottle neck, remove the bottle from the bath, wipe off the exterior of the bottle, and weigh bottle with contents to the nearest milligram. The volume in milliliters occupied by this water at the temperature of the bath is computed from data supplied by the following table (1). The capacity of the bottle at the lowest graduation (zero mark) may be calculated easily, since each minor division equals 0.02 ml.

| Temperature | Apparent Weight of Water in Air |
|-------------|---------------------------------|
| 15° C.      | 0.99805 g./ml.                  |
| 16          | 0.99790                         |
| 17          | 0.99774                         |
| 18          | 0.99756                         |
| 19          | 0.99738                         |
| 20          | 0.99718                         |
| 21          | 0.99697                         |
| 22          | 0.99676                         |
| 23          | 0.99653                         |
| 24          | 0.99629                         |
| 25          | 0.99604                         |
| 26          | 0.99579                         |
| 27          | 0.99552                         |
| 28          | 0.99524                         |
| 29          | 0.99496                         |
| 30          | 0.99466                         |

Knowing the capacity of the bottle at one temperature, we may calculate its capacity at any other temperature, assuming the coefficient of cubical



expansion of the glass to be 0.000025. For example, if the capacity is 45.410 ml. at 20° C., the capacity at 25° C. would be:

$$45.410 [1 + 0.000025 (25 - 20)] = 45.416 \text{ ml.}$$

Changes in the capacity of the graduated neck are negligible for minor temperature changes (10 centigrade degrees or less).

To determine the specific gravity of a liquid, fill the *dry* empty calibrated Babcock bottle with the liquid to any point along the graduated neck. Support the bottle in the constant temperature bath (set for the desired temperature) for 30 minutes, note the reading on the graduated neck, wipe off exterior of the bottle, and weigh bottle with contents. Make no attempt to calculate the weight of liquid needed to fill the bottle to the zero mark. The volume of the liquid in the bottle is simply the zero mark capacity of the bottle at the temperature of the bath increased by 0.02 ml. for each minor division of the reading on the graduated neck. Divide the weight of the liquid in the bottle by its volume to find the apparent weight in air of 1 ml. of the liquid at the temperature of the bath. The specific gravity (apparent) of the liquid at the temperature  $t^\circ$ , referred to water at  $t^\circ$ , is the ratio:

$$\frac{\text{Apparent Weight in Air of 1 ml. of Liquid at } t^\circ}{\text{Apparent Weight in Air of 1 ml. of Water at } t^\circ}$$

The value of the denominator in the above ratio is taken from the table already given.

### Notes

1. Some difficulty may be encountered in drying the empty bottles to constant weight. If water has been used, a rinse with acetone will facilitate drying; but it is important that the residual acetone vapor in the bottle be displaced by air of the same humidity as the atmosphere. Since acetone vapor is about twice as heavy as air, it does not diffuse very readily from an upright Babcock bottle. Hence it is recommended that the bottle be hung with the neck down during drying or that the acetone vapor be removed by suction through a glass tube extending to the bottom of the bottle. Allow the dried bottles to come to equilibrium with the air of the room before weighing.

2. If the liquid is very volatile, a loosely fitting glass cap or plug may be used to minimize evaporation. The cap or plug is weighed with the bottle.

3. The Babcock bottle may also be used for determination of the density (or specific gravity) of a powder by displacement of a liquid of known specific gravity, such as kerosene. In such a determination troublesome air bubbles may be removed by placing the bottle, containing a weighed sample of the powder and a part of the liquid, in a vacuum desiccator and evacuating. Or the mixture may be centrifuged (a Babcock bottle is essentially a centrifuge tube) to remove air bubbles prior to filling the bottle to a point along the neck. The volume to that point having already been determined as indicated above and the weights of sample and kerosene having been noted, the volume of the sample and its density (or specific gravity) are found by rather simple calculations.

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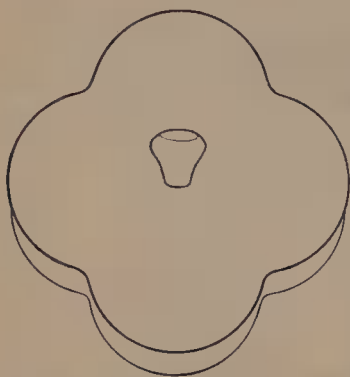


## Baffle Plates for Waring Blendor

E. T. BARTHOLOMEW

University of California Citrus Experiment Station  
Riverside, California

The Waring Blendor is an essential piece of apparatus in the laboratories of many research institutions. Baffle plates for use at different levels in the glass jar of the Blendor have been designed by the writer and found to be very useful.



The baffle plate pictured in the figure was made of  $\frac{1}{2}$ " "Bakelite." Other substances may be used, however, provided they will not absorb any of the components of the materials to be blended.

Since the plate should rest  $\frac{3}{4}$  to  $1\frac{1}{2}$ " above the surface of the materials that are being blended, the size of the plate used in any one operation depends upon the amount of material to be blended. Preferably the edges of the plate should fit snugly against the walls of the jar, but, since the internal dimensions of the jar are not exact, this is not always possible nor is it necessary in order to obtain good results.

Some of the advantages of using baffle plates are as follows: (a) the large-sized glass jar may be used for all operations; (b) a baffle plate of the proper size will confine the material within a restricted area while it is being blended and prevent it from being splashed on or forced up the walls of the jar, where it may be out of reach of the blending assembly; (c) none of the material can be thrown on the lid or the upper portion of the walls of the jar and thus subject to some loss when the operator attempts to recover it with a stream from a washing bottle; and (d) the baffle plate may be washed within rather than above the jar. In research work it is often desirable to recover all the blended material.

---

## Improvised Thermometer Shield

A. DAVIDSOHN

Haifa, Palestine

When using a thermometer as a stirrer, the danger of breakage is very great. Such a thermometer can be protected by placing it in a closely fitting glass tube, somewhat shorter than the thermometer and fire-polished at both ends. The bulb of the thermometer should extend about 2 mm. beyond the lower end of the tube. A short piece of rubber tubing on the upper end of the glass tube holds the thermometer in position. Thus protected, the thermometer may safely be used as a stirrer. The temperature is registered correctly since the bulb of the thermometer is completely surrounded by the liquid.

## Preparation of Filter Paper Pulp

H. F. BRADLEY

United States Smelting, Refining & Mining Company, Midvale, Utah

Filter paper pulp, properly prepared, is superior to some other filter aids for filtering certain materials, such as CdS. One advantage is that it permits the use of a moderately fast paper, which would allow some of the precipitate to run through if used alone but gives a perfectly clear filtrate when pulp is used. One or two milliliters of a moderately thin suspension of pulp, added to the filter just before use, are usually sufficient. In washing the filter, the pulp in the bottom should be disturbed as little as possible.

The following method produces a pulp that is better than most of the pulp sold by dealers. Take 15 to 18 papers (15 cm. diameter), tear into pieces about 1" square and place these in a 600 ml. beaker. Add 300 ml. of dilute sulfuric acid (20 ml. of concentrated acid per 100 ml.) or hydrochloric acid (1:2). Heat to boiling, preferably over a gas flame which heats only the bottom of the beaker. (If a hot plate is used, some of the paper may be carbonized by contact with the hot glass just above the surface of the liquid.) While the mixture is hot, stir until the paper is completely disintegrated and free from lumps (about 5 or 6 minutes). At this point the mixture should have the properties of a rather thin liquid.

Dilute to about 500 ml., filter on a rapid paper (two or more filters may be necessary), and wash free from acid. Sluice the pulp into a beaker with a small amount of water. Small quantities of this thick pulp are diluted with 3 to 5 volumes of water for immediate use.

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## Note on the Combustion of Metallic Calcium

ALEXANDER P. MARION

Queens College, Flushing, New York

The combustion of calcium in oxygen is frequently used to demonstrate the increased speed of the reaction in the pure gas as compared to air. Unfortunately the calcium is usually coated with a layer of oxide which is an extremely poor conductor of heat and may prevent the metal itself from taking fire. Common practice is to scrape as much as possible of the oxide off the calcium turnings before attempting the ignition, but even this is not always effective.

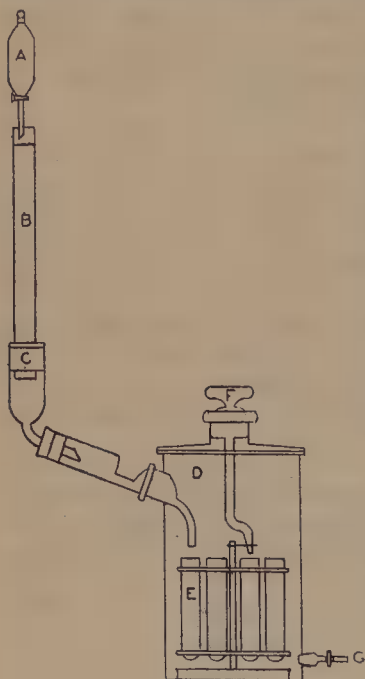
Better results can be obtained if the calcium is subjected to the reducing action of the flame. The metal is heated to near red heat by holding it just above the inner blue cone of the Bunsen flame. Then it is lowered until it is just within the tip of the inner cone and kept there until it is apparently cool. When returned to the hot part of the flame the sample usually ignites, although it may be necessary to repeat the procedure.



## An Apparatus for Columnar Absorption

MERRIAM A. JONES and NOEMIG. ARRILLAGA  
Federal Experiment Station, U. S. Department of Agriculture, Mayaguez, P. R.

The details of a convenient apparatus for columnar absorption are shown in the accompanying sketch. The assembly consists of ordinary laboratory apparatus and its main advantage is that vacuum can be applied continuously while eluted fractions are being collected in separate containers.



The solution to be fractionated as well as the eluant can be run through the separatory funnel, *A*, at a convenient rate into the column, *B*, packed with a suitable absorbent held in place by a plug of cotton or glass wool at the bottom. This plug is in turn held by a piece of screen or hardware cloth cut so that it covers the end and can be held in place by the rubber stopper, *C*. The liquid runs through the adapter and thence to the Brühl receiver, *D*. This piece, a standard catalog item, consists of a set of nine tubes, *E*, mounted on a table which can be rotated by means of an exterior handle, *F*, extending through a ground glass joint. Vacuum is applied at *G*.

Fractions are collected by changing tubes while noting the conditions of the bands in the column. Separate bands can thus be caught in successive tubes.

As a further improvement in the receiver itself, it would be well if the liquid entered through a ground glass joint at *F* and ran through the tube to the receivers. Then the receivers could be stationary and the side entrance to the chamber would not be necessary.

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## Rapid Preparation of Bromine Water

BEN C. SMITH  
Toledo, Ohio

When a solution of bromine in water is used in the quantitative determination of nickel by the colorimetric method, the highest obtainable concentration of bromine is needed for dependable results. It has been the writer's experience that 24-72 hours are usually required for the water to become saturated with bromine.

A sufficiently concentrated solution of bromine can be made quickly by a method analogous to that used in preparing solutions of iodine. An excess of bromine is added to a 5% solution of potassium bromide and the mixture is shaken for about five minutes.

powder. Likewise it appears that the broadest area of application, both wartime and peacetime, is for the technical powder. In this form it lends itself to dilution with an inert extender like pyrophyllite, oil solutions, emulsions, etc. A more restricted field of application requires the aerosol material. This material lends itself for use in the aerosol bomb. Such application requires the D.D.T. to be completely soluble in a specially selected oil and then introduced with "Freon" into a bomb and sealed. By releasing the control on such a bomb, the D.D.T. solution is propelled by the "Freon" gas through an orifice into the air in the form of an aerosol or mist.

The peacetime role of D.D.T. comes into progressively clearer focus with the termination of hostilities in Europe and the Pacific. This war-born chemical seems destined for vital contributions in: (a) agricultural fields, (b) domestic or household application, (c) public health control.

Agriculture will be the greatest economic beneficiary of D.D.T. It is not a panacea for all insect problems. Experimental work thus far reported by the Agricultural Research Administration of the United States Department of Agriculture (3) indicates D.D.T. to be more effective than insecticides now in common use for some thirty pests. Specifically these are the codling moth, cabbage looper, catalpa sphinx, cotton boll worm, cotton flea hopper, Eastern tent caterpillar, elm bark beetle, *Lygus* and four other kinds of sucking bugs, mimosa webworm, pine sawflies, pink bollworm, spruce budworm, velvet bean caterpillar, vetch bruchid, white fringed beetles, mosquitoes, bedbugs, three kinds of lice on man, houseflies and fleas in buildings.

It is further reported that experiments have revealed D.D.T. to be of about equal effectiveness with conventional insecticides in combating the following pests: corn ear worm, diamond back moth, European corn borer, cockroaches, grape berry moth, grasshoppers, imported cabbage worm, oriental fruit moth, potato leaf hopper, silverfish, and the following stored-product pests: cadelle, confused flour beetle, granary beetle, Indian meal moth, lesser grain borer, red flour beetle, rice weevil and saw tooth grain beetle.

Experimental work to date is said to have shown scarcely any effect on the following: the boll weevil, California red scale (adults), cattle grubs, cotton aphid, cotton leaf worm, Florida red scale, Mexican bean beetle, orchard mites, parlatoria scale, plum curculio, red spider, sugarcane aphid and sugarcane borer.

Effective toxicity to insects is but one phase of the overall problem in adapting D.D.T. to agricultural insecticide purposes. Extensive study and investigation must be made by entomologists and insecticide manufacturers in determining these factors:

1. To what extent are plant structures and soil injured by application of D.D.T. insecticides?
2. What tolerance can be established for residual D.D.T. on sprayed or dusted food products (fruits, vegetables and cereals) consumed by humans and livestock?
3. What are the hazards of D.D.T. to wild life, beneficial insects, birds, etc.?
4. Technical formation of D.D.T. insecticides. To what extent will D.D.T. replace totally or partially in insecticidal formulae such materials as the arsenicals, nicotine sulfate, pyrethrum and rotenone? What will be D.D.T.'s compatibility with such components? Can D.D.T.'s effect be synergized? And what is D.D.T.'s most effective particle size?



Thus it is apparent there remains a prodigious amount of experimental work to be performed before competent entomologists and responsible insecticide manufacturers will unqualifiedly release D.D.T. formulated insecticides to the agricultural trade.

The potential applications of D.D.T. in the domestic field seem well indicated also. Above all, its outstanding lethality to the housefly destined it for wide household and barnyard usage. Mosquitoes, bedbugs, roaches, ants, etc., will be among its prime victims. Specially developed and formulated solutions of D.D.T., pyrethrum and other components will in all probability best serve the domestic field. Manually operated spray guns will be in some degree displaced by specially adapted aerosol devices already designed for such application. Domestic animals and pets may gain insect relief through colloidal creams or sprays of low D.D.T. concentration.

Public health will gain an invaluable ally in D.D.T. It promises to be mankind's secret weapon, as a potent destroyer of the mosquito, housefly, louse, tick, etc.—all carriers of the dreaded insect-borne diseases such as malaria, yellow fever, typhus and dysentery. Organized and widespread treatment of insect-infested areas the world over can contribute as never before to the eradication of these ravages.

On occasions in the past we have heard the forebodings of a certain school of gloomy pseudo-scientists, who envisioned the ultimate extinction of man by the insect world. Such fatalistic philosophy now must yield to a growing group of true scientists, who are realistic and enthusiastic in the belief that properly employed D.D.T. or its successors will not only insure man's survival but open up opportunities for new crops, new areas of habitation and cultivation, and new trade routes.

The J. T. Baker Chemical Company is proud to have had an early and prominent part in supplying D.D.T. for the needs of our armed forces. With the coming of peace we look forward to participating in the bright future that lies ahead for D.D.T.

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## MISCELLANY

### Note on Mechanical Stirring

SAUL ROBINSON  
Paterson, New Jersey

When using an electric stirrer to agitate a liquid in a beaker, the latter tends to move and may eventually come into contact with the blade of the stirrer. In most cases movement of the beaker can be prevented or greatly retarded by resting it on three pads of fine sandpaper or emery cloth, made by gluing two small squares together back to back.



# Three important factors\* in the making of products for intravenous injection



- A fine chemical of measured purity
- Your technical skill in manufacturing
- Rigid laboratory control in your plant

Few medicinal products demand as high a degree of purity as that required for products used in intravenous injection.

Upon the absolute *purity* of the intravenous solution, a life may depend.

That is the reason pharmaceutical houses use *extraordinary care* in the making of such products.

In that making, are these three vital factors:

- Selection of a fine chemical known for its purity and uniform quality.

— Extreme care in preparing the intravenous material—in bringing the solution to proper strength, clarifying it, introducing and sealing it into the ampule.

— Maintenance of rigid laboratory control throughout the manufacturing procedure.

The first of these three factors is highly important to manufacturers of *many* types of pharmaceutical products, but *it is even more important to those who produce products for intravenous injections.*

Here, purity to the decimal must be selected *at the beginning*. Your product depends upon that, and your manufacturing job is easier all the way.

*Measured purity* is the foundation upon which Baker has built its service for you. For many

years, leading pharmaceutical and drug companies have known, and bought, Baker's fine chemicals (purity by the ton).

No matter what product you manufacture—if it requires one or more fine chemicals, specify Baker and you'll specify quality *at the beginning*.

J. T. Baker Chemical Co., Executive Offices and Plant: Phillipsburg, N. J.; Branch Offices: New York, Philadelphia and Chicago



**Baker's**  
FINE CHEMICALS



